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3245 North Washington Street

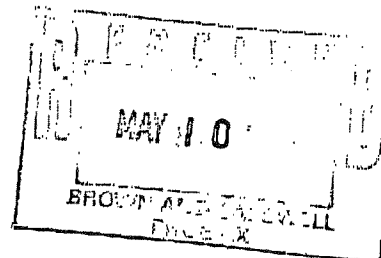
Chandler, AZ 85225

4/29/2005

Sample I.D.

SAMPLE

Quality Assurance Report



Standards

Analysis	Ratio of O/E (O/E \pm 2 σ)	Acceptable limits
Alpha	1.01	0.85 - 1.15
Beta	NA	0.85 - 1.15
Uranium	1.07	0.85 - 1.15
Radon	NA	0.85 - 1.15
Radium-226	0.89	0.85 - 1.15
Radium-228	0.95	0.85 - 1.15
Strontium	NA	0.85 - 1.15
Tritium	NA	0.85 - 1.15

Blanks

Analysis	Observed	Expected	Acceptable
Alpha	<0.5	< 1.0	< 1.0
Beta	NA	< 3.0	< 3.0
Uranium	<0.6	< 0.8	< 0.8
Radon	NA	< 150	< 200
Radium-226	<0.3	< 0.7	< 0.9
Radium-228	<0.4	< 0.7	< 0.9
Strontium	NA	< 0.8	< 0.9
Tritium	NA	< 400	< 500

NA Not applicable.

Robert L. Metzger, Ph.D., C.H.P.

RADIATION SAFETY ENGINEERING, INC.

3245 North Washington Street

Chandler, Arizona 85225

Phone: (480) 897-9459 Fax: (480) 892-5446

May 4, 2005

5/4/05

Date

Robert Z. Meyer

Director's Signature

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I. LAB APPARATUS & REAGENTS.

A. Apparatus.

1. Our apparatus consists of standard glassware for general use.
2. Specialized glassware (and apparatus for use with corrosive materials):
 - a. Multi-distillation unit for tritium (H-3) use, etc.
 - b. De-emanation system for Ra-226 determination.
 - c. Sparging system to degas oxygen from LS cocktails counted by PERALS spectroscopy.
3. Instrumentation.
 - a. Three Tennelec External Gas-Flow Proportional Counters. One Ortec Protean External Gas-Flow Proportional Counters
 - b. One Wallac Model 1414 and One Packard 2700TR Liquid Scintillation Counters.
 - c. Two Intrinsic Ge Crystals and a Canberra Multichannel Analyzer with the Genie Data Reduction Software.
 - d. Four NaI crystals with Multichannel Analyzers for Low Resolution Gamma Spectroscopy.
 - e. Six Surface Barrier Detectors Multiplexed into a Multichannel Analyzer for High Resolution Alpha Spectroscopy.
 - f. Three Photoelectron Rejecting Alpha Liquid Scintillation (PERALS) Spectrometer for Alpha Spectroscopy.

B. Reagents. All reagents used in procedures are of reagent grade or better.

II. LINE OF AUTHORITY.

- A. This line of authority should be used in connection with any laboratory related activities and any administrative matters.
 1. Robert L. Metzger, President
 2. Director
 3. Chief Chemist
- B. This line of authority will be updated or amended as necessary.
- C. All persons listed above are principal quality assurance personnel and responsible for adherence to the quality assurance program.

III. QUALITY ASSURANCE OBJECTIVES.

- A. The objective of this quality assurance program is to produce data that is of known and defensible quality (i.e. the accuracy of the analytical result can be stated with a high level of confidence).
- B. The objectives of the data quality are to produce results that fall under the condition of low bias and high precision which, when combined, express its accuracy. Also, to ensure that the results represent both the sample itself and the sampled population.
- C. The criteria for the laboratory judgement of acceptability of each sample are as follows:
 1. The container must be intact, i.e. no leaks.
 2. The container cap must be intact.
 3. The proper paperwork must accompany it with all pertinent information.

IV. CONTAINER PREPARATION & SAMPLE PRESERVATION SPECIFICATIONS.

A. Sample Containers.

1. All liquid test samples can be collected in plastic (polyethylene or equivalent) containers with the exception of samples to be tested for tritium. Those samples must be collected in **glass containers only**. Zero headspace vials must be used for Rn-222 analysis.
2. Types of Containers.
 - a. 1 pint bottles (polyethylene)
 - b. 1 liter bottles (polyethylene)
 - c. 1 gallon Cubitainers (polyethylene)
 - d. Zero headspace vials (glass)
 - e. 5 or 10 ml vials (glass).
3. Preparation of Containers.
 - a. The containers are clean as provided by the supplier.
 - b. The appropriate preservative, if any (see table 1), will be added so that when the sample is added it will have a pH of less than 2. Any preservative added should be done so at the time the sample is collected unless the sample is to be separated into suspended and dissolved fractions.

Table 1: Sample Container Preservatives

TEST	OPTIONAL PRESERVATIVES
Radon in H ₂ O, H-3, I-131	none
Sr-89/90	HNO ₃
Gross α/β , γ -Spec Samples, Ra-226, Ra-228, Uranium	HCl or HNO ₃

V. DOCUMENTATION ON LAB RECEIVING AND TRACKING OF SAMPLES.**A. Standard methods.**

1. Each sample will arrive at the lab with a chain-of-custody/analysis form.
 - a. This form will contain the following:
 - 1). A unique sample number.
 - 2). Signature of the collector.
 - 3). Date, time and address of collection.
 - 4). Sample type and analysis request.
 - 5). Signatures of persons involved in chain of possession.
 - 6). Inclusive dates of possession.
 - 7). Lab portion includes:
 - a). name of person receiving sample.
 - b). A unique lab sample number and date of receipt.
2. Sample Analysis Request Sheet.
 - a. This will be included in the chain-of-custody form.
 - b. However, a data analysis work sheet will accompany the sample and the chain-of-custody form through the testing procedures.
3. The sample and appropriate sheets will be transferred to the lab as soon as possible.
4. In the lab, the analyzer will inspect the samples' condition, chain-of-custody, assign a lab number, log the sample in the sample log book, and store it in a secure place until analysis.
5. If solids are observed in a sample, the sample will be filtered before analysis.

VI. ANALYTICAL INSTRUMENT CALIBRATION AND FREQUENCY

A. Tennelec Gas-Flow Proportional Counters.

1. Each time a new tank of P-10 counting gas is installed (4-6 weeks) the corresponding instrument is checked for proper operation by counting particle specific check sources.
2. NIST traceable standards are run to determine α/β efficiencies.
3. A Po-210 source is normally used to determine alpha crosstalk in the beta window, however in the absence of Po-210 another isotope can be used and compared to the manufacturers' published data for that isotope. Sr-90 is used to determine beta in alpha crosstalk. Crosstalk is highly energy specific and may not be used by data reduction software.
4. All pertinent information is then entered into the associated computer program which reduces the raw data.
5. To verify the background a blank planchette is counted with each sample run.
6. The α in β cross-talk is checked against manufacturer's values.
7. The β in α cross-talk is checked against manufacturer's values.
8. QC and Tolerances.

A QC is run once a week for both alpha and beta counts. Standards of a known quantity are counted for 30 minutes, then the data is stored and plotted in a database. Warning limits are set at $\pm 10\%$ with action limits at $\pm 15\%$.

B. γ Spectrometer.

1. Energy calibration must be done along with an efficiency calibration for each of the following geometries:
 - a. Planchette - Geometry 1
 - b. 500 mL Marinelli - Geometry 2
2. Calibration is set up initially with the GENIE software and checked monthly with a mixed γ standard in the appropriate geometry (see GENIE manuals).
3. QC and Tolerances.

QC is run monthly to analyze gamma activity of a known standard in one of the three geometries. Error tolerance is $\pm 15\%$ of the standard.

C. Liquid Scintillation Counter.

1. The LS Counters are automatically checked daily by running the calibration rack before each sample run. If the calibration is not successful, the machine will stop automatically.
2. Isotopic calibrations are performed with internal standards with each batch of samples.

D. PERALS®.

1. PERALS® unit is calibrated using NIST traceable spikes with each sample batch.

E. Alpha Spectrometers.

1. The alpha spectrometers are calibrated with NIST traceable electroplated standards of the appropriate geometry for the samples being counted.
2. QC and Tolerances.
QC is done prior to use using a NIST traceable Th-230 standard.
Error tolerance is $\pm 2\sigma$.

VII. PREVENTATIVE MAINTENANCE PROCEDURES & SCHEDULES.

A. General Laboratory Maintenance.

1. A log sheet shall be kept to monitor liquid nitrogen and P-10 counting gas usage.
2. New tanks of liquid nitrogen and P-10 counting gas should be ordered as needed.
3. Wipe tests will be run weekly to determine any contamination of the lab area.

B. Equipment.

1. Perform all calibrations as stated in section VI.
2. Tennelec Gas-Flow Proportional Counter. The pressure of the P-10 counting gas as seen on the flow meter should be at 0.1 and checked daily.
3. High Resolution γ Spectrometers. Check and fill liquid nitrogen dewars weekly (enter into L.N. log-fill sheet).
4. Maintenance of the counting equipment is provided by the manufacturer on an as needed basis. Little preventive maintenance is needed for modern counting equipment. The quality control program tests are adequate to spot equipment problems before they affect data quality.

VIII. ASSESSMENT PROCEDURES FOR DATA ACCEPTABILITY.

- A. Data Reduction and Reporting Procedure: User interactive computer programs which analyze results and propagate error have been developed to prevent error in complex computations. Each program calculates and checks for LLD, reduces the sample data, propagates the error, and reports to the user $< \text{LLD}$ or result $\pm 1\sigma$. These programs may be found in the Procedures manual. If the result indicates the sample contains activity greater than the maximum contamination limit, call DEQ and the client immediately, and arrange for a retest.
- B. Instrument Calibration: The counting instruments are calibrated using NIST traceable standards and recovery through complex chemical processes is determined by spiking samples with known standards. All data is analyzed for accuracy and precision using standard statistical methods.
- C. Laboratory Intercomparisons: This laboratory participates in the ERA InterLab RadChem Proficiency Testing Program. Results of the round robin and performance tests are maintained on file.

IX. CORRECTIVE ACTION PROCEDURES.

If calibration or QC tests fail, notification of the person at the head of the Line of Authority is mandatory, and subsequent investigation of the cause is conducted before proceeding further. Schedule the counting instrument for servicing, if necessary.

X. CHAIN - OF - CUSTODY PROCEDURES.

- A. Chain-of-custody forms are sent out with sample containers and necessary information is filled out by the client/sample holder.
- B. When the sample is returned the date and time received are recorded in the "Received By" box on the chain-of-custody form.
- C. As the sample is treated and tested, the chain-of-custody form accompanies the sample and all results are documented with it.

- D. When all tests have been completed a copy of the chain-of-custody form is mailed along with the report to the client. The original chain of custody form is to be held on file, for each sample, for a period of 3 years. The forms will be maintained in chronological order

XI. REPORTING RESULTS.

- A. All results will be reported in writing to the client.
- B. All reports will be maintained on file by client, for a period of 3 years.

XII. LABORATORY SAFETY.

- A. Licensed environmental laboratories shall comply with all applicable federal, state, and local regulations regarding occupational safety and health.
- B. Radiation safety is maintained under the procedures in our radiation safety program and radioactive materials license (7-192). These documents may be found in the large binders in the lab and in the top file drawer.
- C. Safety equipment.
1. Emergency eye wash stations are placed in obvious and easily accessible locations, above the sink in the lab, and on the reagents' cabinet in the back.
 2. HEPA filters are used in both the glove box and the hood.
 3. The hood face velocity exceeds 100 FPM across the face of the hood.
 4. Geiger and scintillation counters are used to determine the presence of radioactive contaminants in and around the lab area.
 5. Lead bricks and 'L' shields are used in the lab area to protect against γ and β radiation.

XIII. APPROVED ANALYSIS METHODS.

- A. Method 900.0 for Gross Alpha and Gross Beta
- B. Method 600/00-02 for Gross Alpha by Co-Precipitation
- C. Method 903.0 for Total Radium
- D. Method 903.1 for Radium 226
- E. Method 904.0 for Radium 228
- F. Method 913.0 for Radon 222
- G. Method 00-07 - for Combined Uranium
- H. Method 00-07 - for Isotopic Uranium
- I. Method 906.0 - for Tritium
- J. Method 905.0 - for Strontium
- K. Method 901.1 for High Resolution Gamma Spectroscopy
- L. Method 901.1 for I-131

RADIOCHEMICAL DETERMINATION OF THORIUM AND URANIUM IN WATER

Principle

The water sample is filtered. Thorium-234 and uranium-232 tracers are added to 1 to 4 liter aliquots. After evaporation, the uranium is extracted into triisooctylamine (TIOA). The thorium is purified by adsorption on anion exchange resin from nitric acid. Uranium is stripped from the TIOA with nitric acid and coprecipitated. Thorium is stripped from the resin with HCl and coprecipitated. Each actinide is radioassayed by alpha spectroscopy.

Special Apparatus

1. Nuclepore filter membranes, 25 mm dia., 0.2 micrometer pore size or equivalent. See Note 1.
2. Ion exchange column, 2 cm internal diameter x 10 cm.
3. Plastic graduated cylinder, 100 ml volume.
4. Planchets, stainless steel, 32 mm diameter.
5. Separatory funnels, 1 liter capacity.
6. Suction filter for 25 mm membrane.
7. Teflon beakers. See Note 2.
8. Glassware.
9. Pleated filter paper.
10. Alpha spectrometric system consisting of multichannel analyzer, biasing electronics, printer, silicon surface barrier detectors, vacuum pump and chamber.

Reagents

1. Anion exchange resin; BioRad AG1X8 (200-400 mesh, nitrate form) or equivalent. Convert to nitrate form for thorium analysis by washing the resin with 6M HNO_3 until the washing shows no trace of chloride, when tested with AgNO_3 .
2. Ascorbic acid, crystalline reagent.

3. Hydrochloric acid, 12M. 37 percent HCl reagent.
4. Hydrochloric acid, 9M. Dilute 750 ml of the 37 percent reagent grade HCl to 1 liter with distilled water.
5. Hydrochloric acid, 1M. Dilute 83 ml of the 37 percent reagent grade HCl to 1 liter with distilled water.
6. Hydrofluoric acid, 29M, 48 percent HF reagent.
7. Hydrofluoric acid, 3M. Dilute 104 ml of the 48 percent reagent grade HF to 1 liter with distilled water. Use a plastic graduated cylinder and storage bottle.
8. Lanthanum carrier, 0.1 mg La^{+3} /ml. Dissolve 0.0779 g $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ per 250 ml 1M HCl.
9. Nickel foil, 15 cm x 1 cm x 0.1 mm.
10. Nitric acid, 16M, 70 percent HNO_3 reagent.
11. Nitric acid, 6M. Dilute 375 ml of the 70 percent reagent grade HNO_3 to 1 liter with distilled water.
12. Nitric acid, 0.1M. Dilute 6 ml of the 70 percent reagent grade HNO_3 to 1 liter with distilled water.
13. Perchloric acid, 12M, 70 percent HClO_4 reagent.
14. Silver nitrate, crystalline reagent.
15. Silver nitrate, 0.1M. Dissolve 1.7 g AgNO_3 reagent in distilled water. Add 1 ml 6M HNO_3 and dilute to 100 ml with distilled water.
16. Titanium trichloride, 20 percent reagent grade.
17. Titanium trichloride, 0.4 percent. Dilute 1 ml of the 20 percent TiCl_3 to 50 ml with 1 M HCl. Prepare fresh daily.
18. Triisooctylamine (TIOA), reagent grade.
19. TIOA solution in p-xylene, 10 percent. Dissolve 100 ml of triisooctylamine in p-xylene and dilute to 1 liter with p-xylene.
20. p-Xylene, reagent grade.
21. Thorium-234 tracer solution, approximately 800 pCi/ml, accurately calibrated.
22. Uranium-232 tracer solution, approximately 1 pCi/ml, accurately calibrated.

Sample Preparation

1. Filter water sample of one to four liters through a pleated filter.
2. Add 50 ml 12M HCl and measured aliquots of ^{234}Th and ^{232}U tracers.
3. Evaporate sample to 200 ml volume.
4. Add 600 ml of 12M HCl to make sample concentration 9M in HCl.

Uranium Determination

1. Place 100 ml of 10 percent TIOA solution in a 1 liter separatory funnel. Add 50 ml 9M HCl and shake funnel for one minute. Drain and discard lower aqueous acid phase after clean separation of the two phases.
2. Add the aqueous acid sample to the TIOA in the separatory funnel and shake the funnel vigorously for two minutes. Vent the funnel stopcock to prevent pressure buildup in the funnel.
3. Allow the phases to separate cleanly and draw off the lower aqueous acid phase. Save for thorium analysis.
4. Add 50 ml 9M HCl to the TIOA solution in the separatory funnel and shake for one minute.
5. Allow the phases to separate; withdraw and discard lower aqueous acid phase.
6. Repeat steps 4 and 5.
7. Strip the uranium from the TIOA solution by adding 100 ml 0.1M HNO_3 to the separatory funnel and shaking the funnel for two minutes.
8. Allow phases to separate; withdraw and save lower acid phase.
9. Repeat steps 7 and 8 and combine strip solutions. Discard TIOA solution.
10. Place combined strip solutions in the clean separatory funnel.
11. Add 100 ml p-xylene to combined strip solution and shake funnel for one minute. See Note 3.

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12. Allow phases to separate cleanly; withdraw lower aqueous acid layer into beaker. Discard p-xylene.
13. Evaporate combined solution from step 12 to dryness. Do not overheat.
14. Add 100 ml 16M HNO_3 to residue and evaporate to dryness. Do not overheat.
15. Add 5 ml 9M HCl and 5ml 12M HClO_4 to residue and evaporate to dryness.
16. Repeat step 15.
17. Add 10 ml 12M HCl and evaporate to dryness.
18. Repeat step 17.
19. Add 50 ml 1M HCl to sample residue and warm gently to dissolve residue.
20. Heat sample solution to 80°C with stirring. Do not overheat.
21. Suspend clean nickel metal strip into solution for two hours to remove polonium.
22. Remove nickel and evaporate solution to dryness.
23. Add 15 ml 1M HCl to sample residue and warm to approximately 50°C.
24. Add enough ascorbic acid to reduce iron in the sample, indicated by the disappearance of yellow color.
25. Add 1 ml of 0.4 percent TiCl_3 to reduce uranium.
26. Add 1 ml of lanthanum carrier and 5 ml of 3M HF to precipitate LaF_3 carrying uranium. Mix well and set aside for 30 minutes.
27. Using suction, filter coprecipitated sample through a filter membrane.
28. Rinse sample beaker with 10 ml water and add to filter funnel. Rinse beaker with 10 ml ethanol and add to funnel.
29. Remove clamp and top of funnel with suction on. Allow membrane to dry.

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30. Mount membrane carefully on 32 mm planchet using double stick tape.
31. Count sample for 1000 minutes on alpha spectrometer.

Thorium Determination

1. Evaporate aqueous acid fraction containing thorium from step 3 of Uranium Determination.
2. Add 10 ml 16M HNO_3 and evaporate to dryness.
3. Add 5 ml 9M HCl and 5 ml 12M HClO_4 and evaporate to dryness.
4. Add 10 ml of 16M HNO_3 and evaporate to dryness.
5. Repeat step 4.
6. Dissolve sample in 10 ml of 6M HNO_3 with heat.
7. Prepare ion exchange column with 25 ml BioRad AG1X8 resin. Wash resin with 250 ml of 6M HNO_3 .
8. Decant sample into column at gravity flow (approx. 3ml/min) and rinse the sample on the column with an additional 50 ml of 6M HNO_3 . Discard wash.
9. Elute the thorium from the column with 200 ml of 6M HCl at flow rate of 3 ml/minute.
10. Evaporate thorium eluate to dryness.
11. Add 10 ml 12M HCl to residue and evaporate to dryness.
12. Add 15 ml 1M HCl to sample residue and warm to approximately 50°C.
13. Add 1 ml lanthanum carrier and 5 ml of 3M HF . Mix well and set aside for 30 minutes.
14. Using suction, filter coprecipitated sample through a filter membrane.
15. Rinse sample beaker with 10 ml water and add to filter funnel. Rinse beaker with 10 ml ethanol and add to funnel.
16. Remove clamp and top of funnel with suction on. Allow membrane to dry.
17. Mount membrane carefully on 32 mm planchet using double stick tape.

18. Beta count the sample to measure thorium-234 recovery.
19. Count sample for 1000 minutes on alpha spectrometer.

Calculations

Calculate the concentration, Z, of uranium in picocuries per liter as follows:

$$Z = \frac{(A - A_1) \times F}{(2.22)(B - B_1)(E)(V)(T)}$$

where

- A = gross sample counts which appear in the uranium-234,-235, or-238 alpha energy region,
- A₁ = background counts in the same alpha energy region as A above,
- B = gross tracer counts which appear in the alpha energy region of the tracer isotope,
- B₁ = background counts in the same alpha energy region as B above,
- E = alpha detector efficiency,
- F = total calibrated tracer counts for same counting time as sample counts,
- V = sample volume (liters),
- T = counting time (minutes), and
- 2.22 = dpm per pCi.

Calculate the concentration, Z, of thorium in picocuries per liter as follows:

$$Z = \frac{(A - A_1) \times F}{(2.22)(B - B_1)(E)(V)(T)}$$

where

- A = gross sample counts which appear in the thorium -227, -228, -230 or -232 alpha energy region,
A₁ = background counts in the same alpha energy region as A above,
B = gross tracer beta counts,
B₁ = beta counter background,
E = alpha detector efficiency,
F = total calibrated tracer beta counts,
V = sample volume (liters),
T = counting time (minutes), and
2.22 = dpm per pCi.

Calculate the lower limit of detection (LLD) for thorium in picocuries per liter as follows:

$$LLD = \frac{4.66 \sqrt{C_B T}}{(2.22)(E)(R)(V)(T)}$$

where

- C_B = background count rate,
T = counting time (same for sample and background),
E = alpha detector efficiency,
R = fractional yield based on B-B₁/F in calculation,
V = sample volume (liters), and
2.22 = dpm per pCi.

This LLD calculation is valid if the sample counting time is the same as the background counting time.

Notes

1. Nuclepore is a registered trademark of Nuclepore Corp., Pleasanton, CA.
2. Teflon is a registered trademark of Dupont, Co., Wilmington, DE.
3. The p-xylene removes most of the TIOA carried into the aqueous acid phase. Residual TIOA makes the coprecipitation step more difficult.

References

1. Moore, F.L., "Liquid-Liquid Extraction of Uranium and Plutonium from Hydrochloric acid Solution with Tr1 (iso-octyl) amine," Analytical Chemistry 30, 908 (1958).